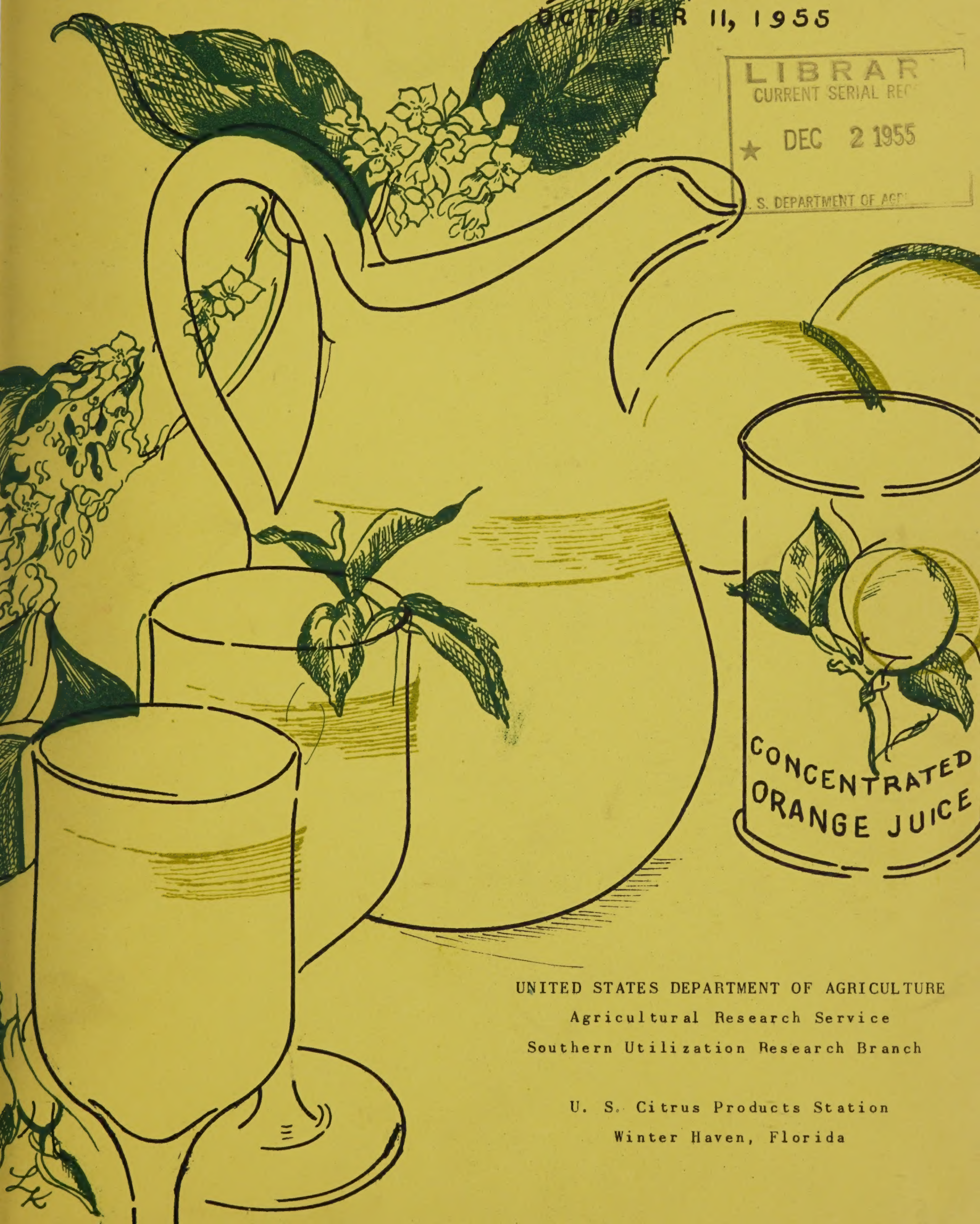
CITRUS PROCESSING CONFERENCE

OCTOBER 11, 1955



UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Service
Southern Utilization Research Branch

U. S. Citrus Products Station
Winter Haven, Florida

LIST OF ATTENDANCE

Fifth Annual
CITRUS PROCESSING CONFERENCE
Florida Room, Citrus Building
Winter Haven, Florida
October 11, 1955

UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Service
Southern Utilization Research Branch
U. S. Citrus Products Station

Altenburger, Michael, Breyer Ice Cream Company, Plant City, Florida
Anderson, E. S., Food Machinery and Chemical Corp., Lakeland, Florida
Atkins, C. D., Florida Citrus Commission, Lake Alfred, Florida
Barron, R. W., Florida Citrus Commission, Lake Alfred, Florida
Bissett, Owen W., U. S. Citrus Products Station, Winter Haven, Florida
Bonnell, J. M., Orange Crystals, Inc., Plant City, Florida
Bradner, Saidee E., Chester C. Fosgate Company, Orlando, Florida
Brent, J. A., Minute Maid Corp., Plymouth, Florida
Bristow, J. J. R., Dunedin, Florida
Brown, J. E., Minute Maid Corp., Plymouth, Florida
Burg, Lilly E., Plymouth Citrus Products Cooperative, Plymouth, Florida
Byer, Ellis M., Florence Foods, Inc., Florence Villa, Florida
Carter, R. D., B. T. Smith Concentrates, Inc., Lakeland, Florida
Chambers, Wm. C., Fosgate Citrus Concentrate Cooperative, Orlando, Florida
Chissom, Gordon A., Corn Products Sales Company, Leesburg, Florida
Conner, J. W., Continental Can Company, Winter Haven, Florida
Cook, R. W., Consulting Engineer, 755 Scotland Street, Dunedin, Florida
Curl, A. L., Western Regional Research Laboratory, 800 Buchanan St., Albany, Calif.
Demsey, J., Continental Can Company, 4645 W. Grand, Chicago, Illinois
D'Ercole, A. D., Birds-Eye Div., General Foods Corp., P.O.Box 481, Lakeland, Fla.
Dougherty, M. H., Florida Citrus Commission, Lake Alfred, Florida
Edwards, G. J., Citrus Experiment Station, Lake Alfred, Florida
Enns, Wm., Continental Can Company, Winter Haven, Florida
Estabrooks, R. G., Minute Maid Corp., Leesburg, Florida
Estes, D. L., Libby, McNeill and Libby, Ocala, Florida
Fisher, C. H., Southern Regional Research Laboratory, 2100 Robert E. Lee Blvd., New Orleans, La.
Garner, R. G., U.S.D.A. - ARS - Office of Experiment Stations Div., Washington, D.C.
Gerwe, R. D., Food Machinery and Chemical Corp., Lakeland, Florida
Gray, L. E., Food Machinery and Chemical Corp., Lakeland, Florida
Greenwood, R. M., Food Machinery and Chemical Corp., Box 75, RFD No. 1, Fort Pierce, Florida
Griffiths, F. P., U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas
Grist, J. D., U.S.D.A. Processed Foods, Winter Haven, Florida
Guyer, R. B., Container Specifications Div., Continental Can Co., Chicago, Ill.

Hamrick, David, Fruit Industries, Inc., Bradenton, Florida
Harding, Paul L., Agricultural Marketing Service, Orlando, Florida
Harvey, H. M., Winter Garden Citrus Products Cooperative, Winter Garden, Fla.
Hendrickson, R., Citrus Experiment Station, Lake Alfred, Florida
Hendrix, J. T., U.S.D.A. Processed Foods, Box 2112, Manatee, Florida
Hill, E. C., Florida Citrus Commission, Winter Haven, Florida
Hinson, W. H., Winter Garden Citrus Products Cooperative, Winter Garden, Fla.
Huggart, Richard, Florida Citrus Commission, Lake Alfred, Florida
Jones, H. L., Brown Citrus Machinery Corp., Winter Haven, Florida
Kesterson, J. W., Citrus Experiment Station, Lake Alfred, Florida
Kew, Theo. J., U. S. Citrus Products Station, Winter Haven, Florida
Kilburn, R. W., Florida Citrus Canners Cooperative, Lake Wales, Florida
Lyle, Mary C., Bordo Products, Winter Haven, Florida
McAllister, Joe, U.S.D.A. Processed Foods, Winter Haven, Florida
McColloch, R. J. Fruit and Vegetable Chemistry Lab., 263 S. Chester, Pasadena 5, Calif.
McDuff, O. R., Adams Packing Assn, Auburndale, Florida
McFarlane, V. H., Southern Regional Research Laboratory, 2100 Robert E. Lee Blvd., New Orleans, La.
McKinnis, R. B., Brown Citrus Machinery Corp., Winter Haven, Florida
McNary, R. R., Florida Citrus Commission, Lake Alfred, Florida
Miller, Don, Pasco Packing Company, Dade City, Florida
Mitchell, W. G., Pasco Packing Company, Dade City, Florida
Moore, E. L., Florida Citrus Commission, Lake Alfred, Florida
Moore, E. P., B and W Canning Company, Groveland, Florida
Morgan, D. A., U. S. Citrus Products Station, Winter Haven, Florida
Murdock, D. I., Minute Maid Corp., Plymouth, Florida
Nanz, Robert, Florida Chemists and Engineers, Inc., 1709 N. Mills, Orlando, Fla.
Newhall, W. F., Citrus Experiment Station, Lake Alfred, Florida
O'Brien, D. J., American Can Company, Tampa, Florida
Olander, J. W., W. F. and John Barnes Co., 301 S. Water St., Rockford, Illinois
Olsen, R. W., Citrus Experiment Station, Lake Alfred, Florida
Packer, W. L., U.S.D.A. Processed Foods, Winter Haven, Florida
Patrick, Roger, Citrus Experiment Station, Lake Alfred, Florida
Petros, L. W., Florida Citrus Canners Cooperative, Lake Wales, Florida
Pobjecky, A. R., Southern Fruit Distributors, Inc., Orlando, Florida
Porges, Nandor, Eastern Regional Research Laboratory, Wyndmoor, Philadelphia 18, Pennsylvania
Pulley, G. N., Snively Groves, Inc., Winter Haven, Florida
Ratcliff, M. W., Florida Citrus Canners Cooperative, Lake Wales, Florida
Rouse, A. H., Citrus Experiment Station, Lake Alfred, Florida
Rushing, N. B., U. S. Citrus Experiment Station, Winter Haven, Florida
Sampson, G. O., American Can Company, Tampa, Florida
Schwartz, S. J., U.S.D.A. Processed Foods, 524 Minn. St., Lake Wales, Florida
Scott, W. C., U. S. Citrus Products Station, Winter Haven, Florida
Senn, V. J., U. S. Citrus Products Station, Winter Haven, Florida
Shea, K. G., O.Q.M.G., 2nd and T Street SW, Washington, D. C.
Siconolfi, U.S.D.A. Processed Foods, Winter Haven, Florida
Simon, Morris, Fruit and Vegetable Products Div., QMC, 1819 W. Pershing Road Chicago 9, Illinois
Singleton, Gray, J. Wm. Horsey Corp., Plant City, Florida

Smith, F. C., 1231 - 13th St., NW, Winter Haven, Florida
Spross, S. A., J. Wm. Horsey Corp., Plant City, Florida
Stivender, F. H., Food Machinery and Chemical Corp., Lakeland, Florida
Swift, L. J., U. S. Citrus Products Station, Winter Haven, Florida
Ting, S. V., Florida Citrus Commission, Lake Alfred, Florida
Todd, H. D., Florida Citrus Canners Cooperative, Lake Wales, Florida
Tourjee, E., Food Machinery and Chemical Corp., Lakeland, Florida
Trumm, Howard, Libby, McNeill and Libby, Ocala, Florida
Veldhuis, M. K., U. S. Citrus Products Station, Winter Haven, Florida
Vilece, R. J., Dept. of Hort., Univ. of Florida, Gainesville, Florida
Walker, S. S., Thornton and Company, Box 2880, Tampa, Florida
Wenzel, F. W., Citrus Experiment Station, Lake Alfred, Florida
Westbrook, G. F., Florida Dept. of Agriculture, Winter Haven, Florida
Winoker, J., Food Machinery and Chemical Corp., Lakeland, Florida
Wolford, R. W., Florida Citrus Commission, Lake Alfred, Florida

PROGRAM AND ABSTRACTS OF PAPERS

FIFTH CITRUS PROCESSING CONFERENCE

October 11, 1955

Florida Room, Citrus Building
Winter Haven, Florida

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ORGANIZATIONS PARTICIPATING
in
FIFTH CITRUS PROCESSING CONFERENCE

SOUTHERN UTILIZATION RESEARCH BRANCH
Citrus Products Station, Winter Haven, Florida
Fruit and Vegetable Products Laboratory, Weslaco, Texas

WESTERN UTILIZATION RESEARCH BRANCH
Fruit and Vegetable Chemistry Laboratory, Pasadena, Cal.
Western Regional Research Laboratory, Albany, Cal.

EASTERN UTILIZATION RESEARCH BRANCH
Eastern Regional Research Laboratory, Philadelphia, Penna.

CONTINENTAL CAN COMPANY, INC., CHICAGO, ILL.*

* Under a Memorandum of Understanding conducts cooperative research on the heat treatment of citrus juices with the United States Department of Agriculture at the U. S. Citrus Products Station, Winter Haven, Florida.

PROGRAM
of
CITRUS PROCESSING CONFERENCE
October 11, 1955

MORNING SESSION

Chairman: M. K. Veldhuis, In Charge
Citrus Products Station
Winter Haven, Florida

9:30 A.M. Opening Remarks

12:00 Recess for lunch

AFTERNOON SESSION

Chairman: Vernon H. McFarlane
Fruit and Vegetable Section
Southern Utilization Research Branch
New Orleans, Louisiana

GROWTH RATES OF LACTOBACILLUS AND LEUCONOSTOC SPECIES IN ORANGE CONCENTRATES

N. B. RUSHING, M. K. VELDHUIS AND V. J. SENN
Citrus Products Station
Winter Haven, Florida

Certain species of bacteria are known to be capable of causing the type of spoilage of orange concentrates known as "buttermilk off-flavor". Growth rates were determined for two strains each of Lactobacillus brevis, L. plantarum, var. mobilis, Leuconostoc dextranicum and L. mesenteroides which had been isolated from commercial concentrates exhibiting this type of spoilage. These experiments consisted of two parts: the first part was concerned with the influence of pH, and the second part with the concentration of orange juice on the growth rates. In the first part of the study growth rates were determined for 12° and 18° Brix orange juices at pH values of 3.4, 3.6, 3.8, and 4.0. In the second part growth rates were obtained for concentrations of orange juice at 12°, 18°, 24°, 32°, 37°, and 42° Brix adjusted to pH 3.8. A report on the growth rates of these organisms at 12° Brix and pH 3.4, 3.6, 3.8, and 4.0 was given last year and is not included in this discussion.

Growth rates of all species decreased with increasing concentration and increased with increasing pH. The effect of concentration was roughly parallel for all species, but marked differences were observed in response to pH variations. The lactobacilli were less sensitive than either of the Leuconostoc species, and L. mesenteroides was far more affected by pH change than were any of the others. L. mesenteroides was killed slowly at high acidity or high concentration and L. brevis at high concentration. There was no significant difference in response between strains of the same species. The results of this investigation are summarized in Table I.

Generation times are given in Table II. The shortest generation time found in the entire series of studies was 1.4 hours for L. mesenteroides in 12° Brix juice at pH 4.0. Because generation times were so long it is believed unlikely that off-flavor due to growth of these organisms can develop in a commercial concentrator unless static pockets or films of juice exist.

[illegible] RATES OF [illegible]
IN [illegible]

[illegible], M. L. [illegible]
Citrus Products [illegible]
Winter Haven, Florida

[illegible] species of [illegible] to [illegible] of [illegible] age of [illegible] as "[illegible]" [illegible] in the [illegible] of [illegible] *[illegible]cium* [illegible] trate[illegible] parts: the first part [illegible] the second part [illegible] on the growth [illegible]. In the [illegible] determined for 10° and 1[illegible]° [illegible] values of [illegible], [illegible], and 4.0. In the second part [illegible] orange [illegible] pH [illegible] a report on the growth [illegible] 3.6, [illegible], and 4.0 [illegible] not included in this discussion.

[illegible] with [illegible] pH. [illegible] for [illegible] differences [illegible] species, and *[illegible]* [illegible] were [illegible] killed [illegible] species. [illegible]

[illegible] are [illegible] in Table [illegible] species [illegible] second [illegible] due to [illegible]

Table 1 - Effect of pH and concentration of orange juice on the growth rates of four species of bacteria.

Orange juice degrees Brix	pH	Lactobacillus brevis strain no. B27	B28	Lact. plant. var. mobilis strain no. B29	B32	Leuconostoc dextranicum strain no. B34	B35	Leuconostoc mesenteroides strain no. B42	B47
12	3.8	.077	.079	.094	.093	.137	.137	.131	.132
18	3.4	.027	.028	.035	.036	.033	.033	-.037	-.035
	3.6	.038	.037	.047	.044	.051	.056	-.025	-.024
	3.8	.067	.063	.076	.073	.089	.09	.081	.081
	4.0	.086	.086	.098	.095	.114	.111	.10	.10
24	3.8	.054	.054	.065	.062	.071	.073	.065	.067
32	3.8	.031	.037	.048	.048	.042	.044	.037	.037
37	3.8	.0057	.0153	.025	.023	.023	.029	.024	.022
42	3.8	-.021	-.026	.014	.012	.014	.014	-.006	-.003

Table 2 - Generation times in hours of Lactobacillus and Leuconostoc species in orange juice or concentrates at various pHs.

Orange juice degrees Brix	pH	Lactobacillus brevis	Lactobacillus plantarum var. mobilis	Leuconostoc dextranicum	Leuconostoc mesenteroides
12	3.8	3.9	3.6	2.2	2.3
18	3.4	10.7	8.4	9.1	*
	3.6	7.9	6.7	5.6	*
	3.8	4.6	4.0	3.3	3.7
	4.0	3.5	3.1	2.7	3.0
24	3.8	5.6	4.7	4.2	4.6
32	3.8	8.8	6.3	7.0	8.1
37	3.8	27.	12.5	11.6	13.1
42	3.8	*	23.	22.	*

* Negative growth rates were encountered under these conditions.

√ THEORY AND PRACTICE OF DAIRY WASTE TREATMENT BY OPTIMAL AERATION

NANDOR PORGES
Eastern Regional Research Laboratory
Philadelphia 18, Pennsylvania

A treatment for the disposal of dairy waste developed at the Eastern Regional Research Laboratory has proven to be of considerable interest to the dairy industry and might also be of interest to the citrus industry. The treatment accelerates the natural process of converting organic polluting substances to harmless material. It utilizes the oxidative activity of microscopic life feeding upon organic matter present in dairy waste requiring oxygen to maintain this life activity. Sufficient air is supplied in order that adequate amounts of oxygen are available when needed.

Laboratory studies showed that adequate aeration and rigorous agitation prevent odor and acid formation in the complete treatment of 1000 ppm dairy waste. One pound of available oxygen in solution is required to oxidize waste with an oxygen demand of one pound. At first the waste is removed from solution and assimilated. During this assimilation phase about 37.5% of the total oxygen is utilized in the short span of 3 hours by 1000 ppm sludge, producing 500 ppm new sludge. The sludge also oxidizes itself at the rate of one percent of its own weight per hour by endogenous respiration. The rate of oxygen utilization is very low in this phase.

Equations based upon the chemical composition of the waste and sludge are given to show these changes.

In general:

$$\text{Organic wastes} + O_2 \xrightarrow{\text{assimilation}} \text{cells} + CO_2 + H_2O$$

$$\text{and cells} + O_2 \xrightarrow{\text{endogenous resp.}} CO_2 + NH_3 + H_2O$$

Specifically for milk wastes:

$$\underset{\text{(sugar)}}{8\ (CH_2O)} + \underset{\text{(protein)}}{C_8H_{12}N_2O_3} + 6\ O_2 \longrightarrow 2\ \underset{\text{(cells)}}{C_5H_7NO_2} + 6\ CO_2 + 7\ H_2O$$

$$\underset{\text{(cells)}}{C_5H_7NO_2} + 5O_2 \longrightarrow 5\ CO_2 + NH_3 + 2\ H_2O$$

The data have been applied in the development of treatment units on pilot plant and industrial size. A research project sponsored by U.S.D.A. at the Pennsylvania State University successfully treated 10,000 gallons of waste daily from the University Creamery. Milk solids were oxidized without odor when sufficient oxygen was available. After evaluating a number of non-mechanical proprietary aerating devices, Professor of Sanitary

Engineering, R. R. Kountz, selected a Penberthy Ejector, type XL-96, size 7A (steam nozzle) to supply oxygen in solution by direct aspiration from the atmosphere. Excess sludge was no problem since endogenous respiration destroyed 20% of the sludge daily. Conditions were established wherein the new cells produced from the milk wastes replaced the amount lost by endogenous respiration.

A fill-and-draw commercial unit was designed and put into operation by Prof. Kountz to handle 25,000 gallons waste daily with a C.O.D. of 2000 ppm. A 37,500 gallon tank with an effluent pipe at the 9,000 gallon level is used. Aeration is stopped for 6 hours to allow cells to settle and to drain out the 25,000 gallons clear supernatant. The sludge concentration is 5,500 ppm, giving a total of 750 pounds of sludge solids. Endogenous respiration required 9.4 pounds oxygen per hour to oxidize 150 pounds cells. During the 8 hours of waste flow, 17 pounds of oxygen are required per hour in the assimilation phase for the conversion of the 300 pounds of milk solids in the waste. Thus 26.4 pounds oxygen are needed per hour for the first 8 hours. The 150 pounds of cells that are produced replace those oxidized by endogenous respiration.

The one-tank fill-and-draw process is practically automatic and if left undisturbed gives 95% removal of B.O.D. and produces no disagreeable end products. A continuous process incorporating these principles is under development.

COLOR ANALYSIS OF RUBY RED GRAPEFRUIT

B. J. LIME, T. S. STEPHENS, AND FRANCIS P. GRIFFITHS
Fruit and Vegetable Products Laboratory
Weslaco, Texas

Color comparison and analysis of fruit from four groves of Ruby Red Grapefruit were made at two week intervals during the 1954-55 grapefruit season. Color measurements on 30 cut surfaces of halved fruit were made with a Gardner Automatic Color Difference Meter, using the small illumination opening. Four segments were removed from each half of each 30 fruit sample; blended, deaerated, and the color and reflectance of the blended sample measured. Brix, acid, and naringen values were also determined.

Carotene and lycopene of the blended samples were determined by two methods. (1) A standard procedure which separates the two pigments chromatographically, and (2) a shorter method which measures the absorption of a hexane extract at wave length 455 mu (maximum for carotene) and 505 mu (maximum for lycopene). From these values total pigmentation in terms of lycopene and carotene is calculated by use of simultaneous equations.

Results obtained by the short method are about 40% higher for lycopene and 20% for carotene because closely related pigments are not removed on a chromatographic column as is done in the longer procedure. The ratio of a (redness) over b (yellowness) determined by the Gardner Automatic Color Difference Meter, shows close correlation with the ratio of lycopene over two times carotene ($1/2c$) obtained by the short procedure.

Color differences between individual fruit were greater than measured differences of composite samples. Composite samples of fruit from two groves on sandy loam soil were slightly more colored than samples of fruit from two groves on heavier clay soil. Seasonal color variations in fruit paralleled that observed and reported previously, maximum color existing prior to maturity, decreasing sharply during December, January, and February, and continuing to fade slightly during the remainder of the season.

It is concluded that the ratio of a over b as measured by the Gardner Automatic Color Difference Meter on blended samples of fruit pulp provides a reproducible index of color intensity.

COMPOSITION OF HAND REAMED JUICE, COMMERCIALLY EXTRACTED JUICE, AND PEEL JUICE OF ORANGES AT WEEKLY INTERVALS DURING THE 1954-55 SEASON

LYLE JAMES SWIFT
Citrus Products Station
Winter Haven, Florida

Excessive extractor finisher pressures are generally considered to be related to gelation and clarification problems in orange concentrates, but little is known of other possible effects such as the influence on flavor, titratable acidity, pH, density, and soluble solids as well as on the contents of sugars, soluble pectic substances, flavonoids, etc. The present study was undertaken to supply some of this information and, if possible, to devise a test for the amount of peel extract in citrus juices.

For the collection of samples a concentrate plant was chosen in which the peel juice passes directly from the Pipkin presses to the oil centrifugals without dilution with water. In addition to centrifuge effluent, samples of the commercial orange juice were always taken as well as oranges from which the juice was later carefully expressed by hand. All samples were collected at weekly intervals throughout the season and were frozen and stored until the collection was complete. At this time the samples were thawed, filtered by suction with Hyflo-Supercel, resealed in 4-oz. cans, and refrozen until they were withdrawn for analysis.

Titratable acidity was always lowest in the peel juices and usually highest in the hand-pressed juices with the commercial juices usually falling in between which might be indicative of peel juice contamination.

Soluble pectic substances were always highest in the peel juices. Except for a few samples taken early in the season, these substances were higher in the commercial juices than in those that were hand pressed.

The diacetyl was always highest in the peel juices and was usually somewhat higher in the commercial juices than in the hand pressed. Diacetyl in all types of juices rose steeply at the end of the season.

Both reducing and non-reducing sugars trended upward as the season progressed. This was true in the peel juice as well as in the commercial and hand-pressed juices. In general, the reducing and non-reducing sugars were present in about equal amounts in all the types except early season peel juice where the reducing sugars averaged about 50% higher than the non-reducing sugars.

PRELIMINARY STUDIES ON THE RELATIONSHIPS BETWEEN REFRACTIVE INDEX, SOLUBLE SOLIDS CONTENT, AND DENSITY OF CITRUS JUICES AND CONCENTRATES

DONALD A. MORGAN AND W. CLIFFORD SCOTT
Citrus Products Station
Winter Haven, Florida

Industry advisory committees have pointed out the need for improvement in the accuracy of refractometric determinations of soluble solids in citrus concentrates. Preliminary investigations at the Station have indicated the diversity and magnitude of problems involved, and these are reported here.

Temperature corrections are not included in this study, as facilities are available for conducting investigations on effects of other variables at a constant temperature of 20° $\pm$ 0.05° C.

Refractometer scales in use are based on the refractive index of solutions of pure sucrose. The only correction commonly applied for normal constituents of orange juice products is for citric acid. Stevens and Baier, in 1939, determined the refractive index and Brix of solutions of known citric acid content, and prepared tables of correction factors for refractometer readings. They recognized, but dismissed as relatively unimportant, the effect upon refractive index of invert sugar, ash, insoluble solids and essential oils.

Citric acid is a tri-basic acid which, in distilled water, has a pH of 2.6. The pH of orange juice indicates partial neutralization in vivo, with only about two-thirds of the citrate content being titratable acid. Fortunately, the refractive index of potassium citrate is very close to that of sucrose and its presence in expected amounts should result in refractometer readings for 42° Brix concentrate about 0.1° Brix too high.

Since approximately half of the sugars in citrus juices are invert, it appears that refractive index-sucrose tables should not be used without modification. Available information (Browne and Zerban) indicates that the refractive index of glucose is very close to that of sucrose, while the refractive index of fructose is appreciably lower. In orange juice, fructose constitutes about one-fourth of the total sugars present. Calculations based on existing tables indicate that this constituent should cause refractometers to read about 0.25° Brix too low in indicating the total soluble solids content of 42° Brix orange concentrate.

An important constituent of citrus juices which causes trouble in the refractometric determination of soluble solids is the pulp, or insoluble solids. The refractometer is influenced only by the solids dissolved in the aqueous phase of the mixture, and therefore gives an erroneously high reading. For example, consider a sample of concentrate containing 42 grams soluble solids, 2 grams insoluble solids, and 56 grams of water. This mixture contains 42% soluble solids, but the aqueous phase contains 42/98, or 42.85%, soluble solids.

Powdered orange juices are being used in studying the effects of various constituents on refractive index. These powders are prepared in the laboratory vacuum shelf drier from citrus juices of different varieties and maturities, and include wide variations in pulp and acid content. By determining moisture and insoluble solids content, soluble solids can be calculated accurately. Refractometer readings and density determinations were made on samples reconstituted to about 55°, 42°, and 12° Brix.

Efforts to achieve the high order of accuracy needed have been fraught with difficulties. Reading of even the Precision refractometer is difficult because of the light scattering effect of citrus concentrates. Slight errors in the determination of moisture by the Karl Fisher method in citrus powder can introduce a serious error in the preparation of samples of known soluble solids content. Citrus powders are very hygroscopic and all transfers must be made in a low humidity cabinet. Extreme care must be taken to avoid loss of soluble solids during reconstitution, and to ensure a perfectly homogeneous concentrate free of air.

These investigations have been underway only a short time. To date, efforts have largely been directed toward overcoming many of the difficulties mentioned above. Sufficient data has been secured to indicate that errors arising from the heterogeneous nature of citrus juices are important, but a great deal more data must be obtained and analyzed before correction factors can be recommended.

MECHANISMS OF CLOUD LOSS IN FROZEN ORANGE CONCENTRATES

R. J. McCOLLOCH
Fruit and Vegetable Chemistry Laboratory
Pasadena 5, California

Studies on the effect of heat treatment on pectinesterase (PE) activity and on cloud stabilization conducted at the Pasadena Laboratory, and confirmed by the work of other laboratories, have demonstrated a notable lack of correlation between PE activity and cloud stability. These and other studies have led to a re-examination of the known facts concerning the properties and activity of citrus PE. A survey of the literature reveals that there is at present nothing better than circumstantial evidence linking PE activity to cloud loss. The only available studies of the properties of this enzyme have been conducted in vitro under conditions which make it unsafe to extrapolate the information obtained to the medium of orange concentrates.

Consequently studies have been initiated at Pasadena to determine factors in orange concentrates which affect cloud stability and to relate the effect of these factors on cloud stability to a corresponding effect of PE activity. As a result of these studies it can be shown that the characteristic variation of cloud stability with concentration in orange juice is largely governed by concomitant variations in sugar and citrate concentrations. However, sugar and citrate tend to inhibit PE activity and appear to produce maximum inhibition at the concentration level where minimum cloud stability is observed. These and related observations have made it appear that PE activity is not the principal factor in determining cloud loss.

Investigations of other possible mechanisms of cloud loss have, therefore, been carried on. As a result several other hydrolytic enzyme systems have been found to bring about varying degrees of cloud loss. Of greater interest, however, are results which appear to implicate biological oxidation-reduction systems in cloud loss. Studies based on the supposition that oxidation-reduction systems may be involved in cloud loss have already produced results of practical value. In general cloud loss in orange concentrates is found to be accelerated by compounds which are good proton-donors. Some of these compounds, such as glutathione and various phenolic compounds, occur naturally in orange juices. Many compounds which can act as proton-acceptors tend to inhibit cloud loss. The most notable of these is fumaric acid, a natural constituent of most food materials and an important intermediate in biological oxidation-reduction systems. Both fumaric acid and its simple salts bring about 50% or greater inhibition of cloud loss in California orange concentrates when added at the 0.025 molar level.

Studies are continuing to unravel the apparently complex system of enzymatic and non-enzymatic mechanisms of cloud loss. Judging by results already obtained, it seems not unreasonable to assume that a thorough understanding of the mechanism of cloud loss may lead to chemical methods of completely stabilizing cloud without resort to heat treatment.

A STUDY OF SEASONAL AND VARIETAL FACTORS AS RELATED TO HEAT STABILIZATION OF FROZEN ORANGE CONCENTRATE

R. B. GUYER AND W. M. MILLER
Metal Division Research & Development
Continental Can Co., Inc.
Chicago, Illinois

O. W. BISSETT AND M. K. VELDHUIS
U.S.Citrus Products Station
Winter Haven, Florida

During the past three Florida citrus seasons, the Continental Can Company and the U.S.D.A. have conducted a cooperative project at their Citrus Products Station in Winter Haven, Florida. This project was undertaken to study heat stabilization of frozen orange concentrate and has included investigations of heat stabilization time and temperature studies, the effect of heat treatment at various stages of concentration, a comparison of several methods of heat stabilization, the stability of high Brix concentrate, and a study of varietal and seasonal changes as related to heat stabilization. The following report deals with the latter phase of the investigation.

This study was carried out during the 1952-53 season on Pineapple and Valencia varieties and during the 1953-54 season on the Hamlin, Pineapple, and Valencia varieties. For the purpose of this study, oranges were picked either once a week or once every two weeks from selected groves and trees. In this way each harvest date represented a change in maturity since the oranges came from the same trees at each picking. The juice was extracted and heat stabilized at either 150°, 160°, 170°, 180°F. for 5 seconds in a small tube turbulant flow heat exchanger. The heat-treated juice was concentrated to 55° Brix in a pilot plant evaporator and was cut back to 42° Brix with unheated single strength juice.

The results indicated little or no effect of maturity on enzyme inactivation for Hamlin and Pineapple varieties. However, it was noted that heat treatment at 150°F. and 160°F. in the early season for the Valencia variety was more effective than later in the season.

In comparing the cloud stability of the three varieties, it was noted that concentrate made from Valencia orange juice was more stable than concentrate made from either Pineapple or Hamlin juice. In turn, the concentrate made from Pineapple juice was somewhat more stable than that made from Hamlin juice.

In comparing the effect of various heat treatments on cloud stability, it was noted that heat treatment of juice from Hamlin oranges even at 180°F. retarded cloud loss for only seven days when stored at 40°F. Pineapple orange juice heat stabilized at temperatures up to 180°F. remained stable for 14. days. On the other hand, Valencia concentrate variables remained stable for much longer periods with heat treatment of 150°F. or 160°F. for 5 seconds.

No significant difference was noted concerning the effect of packing dates on cloud stability for the Hamlin and Pineapple varieties. Concentrates made from early season Valencia fruit heat-treated at 150°F. or 160°F. showed greater response to the heat treatment than concentrate made from late season fruit given the same treatment.

THE PRODUCTION AND STABILITY OF "HI-BRIX" FROZEN ORANGE CONCENTRATE (58.5° Brix).

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Cooperative investigations during the past three seasons between the Continental Can Company, Inc., and the U.S.D.A. (Citrus Products Station) have been concerned with the heat stabilization of frozen concentrated orange juice. They have been divided into the following projects:

1. Effect of heat treatment on enzyme inactivation, cloud stabilization, and flavor.
2. Effect of varietal and seasonal changes.
3. Effect of heat treatment at intermediate stages of concentration.
4. Evaluations of methods of heat stabilization.
5. Production and stabilization of 5 plus 1 concentrate.

Results of only the last project are reported here.

A sufficient quantity of Valencia oranges were harvested at one time to supply all the juice used in this study. They were stored at 40°F., and boxes removed in randomized manner for each day's run. Juice was extracted each day, screened and held at 40°F. in a coldwall tank until used. When heat treatment was used, it was applied either with a small-tube turbulent-flow or a commercial plate-type heat exchanger.

This investigation was divided into three parts. The first was designed to compare the relative stability of 42° Brix and 58.5° Brix concentrates. Unheated juice and juices heated to 150°, 160°, 170°, and 180° F. were used in the study. The 42° Brix concentrates were prepared by concentrating to 55° Brix and cutting back with unheated juice. The 58.5° Brix samples were prepared by concentrating to 68° Brix and cutting back with unheated juice. Comparisons were made at stated intervals during six weeks of storage at 40° F. Tests included residual enzyme activity, cloud stability, and flavor. There was no appreciable difference in the residual pectinesterase between products receiving similar heat treatments. The high-Brix concentrates were inherently more stable than those of 42° Brix. Heat treatment of the 42° Brix products at 150° F. resulted in cloud stability approximately equal to that of unheated high-Brix concentrates, while heat treatment at 150° F. was sufficient to impart cloud stability for the entire 42 days to the 58.5° Brix products. Residual pectinesterase activity and cloud stability data did not indicate any difference in the effectiveness of the two heat exchangers used.

The second part of the experiment was designed to find whether high-Brix Valencia concentrate could be stabilized by heat treating only a portion of the evaporator feed juice. Unheated juice and juices heated to 150°, 160°, 170°, and 180° F. were respectively concentrated to 68° Brix. Unheated and heat-treated concentrates were then blended in six different ratios. One part of heat stabilized concentrate added to three parts unheated concentrate greatly improved the stability over the control, while a 50-50 mixture provided complete stability for the entire 42 days. No advantage was demonstrated by treatment of the evaporator feed juice at temperatures above 150° F.

The third part of the experiment was designed to determine the optimum ratio of concentrate to cutback juice in the preparation of high-Brix concentrate. Both heated and unheated portions of juice were concentrated to 62°, 64°, and 66° Brix and cut back to 58.5° Brix with unheated juice. Unheated controls were stable for 9 days at 40° F., while all products containing heated juice were stable for the entire 42 days of observation.

Flavor comparisons were made between 42° and 58.5° Brix concentrates prepared from juice stabilized at the various temperatures with both the small tube and the plate heat exchangers. No differences could be detected between the degrees of concentration, the temperatures used, or the two types of heat exchangers.

CHANGES IN THE CAROTENOID PIGMENTS OF VALENCIA ORANGE JUICE DURING CONCENTRATION, POWDER PREPARATION, AND STORAGE OF POWDER

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The off-flavor which develops in canned orange juice or powder on prolonged storage may originate, at least in part, in the carotenoid pigments. The carotenoids of California Valencia orange juice were previously investigated. Countercurrent distribution in a glass Craig apparatus was employed to separate the carotenoids into six fractions--hydrocarbons, monols, diols, monoether diols, diether diols, and polyols. These fractions were then resolved by chromatography into 26 components, not counting cis-isomers which may be artifacts. Five of these contained epoxide groups, 9 furanoxide groups and two both groups. These cyclic ethers amount to over half of the carotenoid content of orange juice.

The carotenoids were investigated in a similar manner in samples of frozen single-strength and concentrated juices prepared from Florida Valencia oranges. The total carotenoid content of the Florida Valencia juice was somewhat lower than that found in California Valencia juice, but the qualitative and quantitative composition of the pigment mixtures were quite similar. Three minor constituents were found in the Florida juice which have not yet been found in California juice. There was no significant difference between the composition of the carotenoid mixtures from the frozen single-strength and concentrated Florida juices.

Powdered juice was prepared by the puff-drying process from the concentrate to which had been added additional suspended material (which includes the carotenoid pigments) from single-strength juice. Examination of the carotenoid fraction of the powder showed that a minor part of the carotenoid epoxides had been isomerized to furanoxides. This acid-catalyzed isomerization results in paling of the color since the furanoxides absorb light at shorter wave-lengths than the corresponding epoxides. There was no apparent change in the non-ether carotenoids.

The powdered juice was conditioned for 78 days at 77°F. in order to reduce the moisture content to the desired level. Examination of the carotenoids at this time showed that over half of the epoxides had been converted to the isomeric furanoxides. There were no significant changes in the non-ether carotenoids. Included in the latter are the provitamins A, cryptoxanthin, β- and α-carotenes. The conditioned powder is now undergoing storage for six months at 100°, following which it is planned to examine the carotenoids again.

The carotenoid epoxides are mild oxidizing agents. Reactions involving this property may result in substances causing off-flavor, either from the carotenoids themselves or from the accompanying lipides.

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